EFFECTS OF ANNEALING ON THE MAGNETIC PROPERTIES OF COLD-WORKED NICKEL-ZINC FERRITE

Sixth Technical Report

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Honeywell Corporate Research Center

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36

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13. ABSTRACT

Magnetic property changes in nickel-zinc ferrite toroids caused by polishing are shown to be recoverable during annealing at temperatures in the range of 500 to 1000°C.

Recovery is shown to be an athermal process, leading to the speculation that internal strains may be due to coherency effects between stacking faults and the parent ferrite lattice, and that stress-relief under those conditions may not be independent of the presence of a second phase.

Some consideration is given to the possibility of using recovery effects to indicate maximum temperature exposure in some type of sensor, but it is concluded that a great deal more work would be needed to develop such an instrument.

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TABLE OF CONTENTS

I. INTRODUCTION

In an earlier report[1] it was shown that the magnetic properties of a nickel-zinc ferrite were markedly degraded by cold-work, and that the effects could be understood in terms of residual elastic stresses together with a stress-induced phase transformation. It was also noted that the magnetic property changes could be reversed by annealing, though no quantitative data were presented.

It is the purpose of the present report to evaluate the effects of annealing time and temperature on the magnetic properties of ferrite samples of varying thickness (and hence varying degrees of cold-worked surface material in relation to total volume).

II. EXPERIMENTAL METHODS

A. FABRICATION OF TOROIDS

Toroidal samples were prepared from sintered blocks of $Ni_{.36}Zn_{.64}Fe_2O_4$ ferrite by cutting thin slices from the blocks and then ultrasonic machining. Sample dimensions were fixed at 0.44 inch O.D. x 0.18 inch I.D., while thicknesses were chosen to be 0.004 inch, 0.008 inch, and 0.018 inch. Final sizing of the samples was done by polishing with 16μ diamond dust on a teak lap.

B. ANNEALING TECHNIQUE

As-polished toroids, similar to those whose magnetic characteristics were well established in earlier work[1], were annealed at temperatures from 500 to 1100°C for times of 20 minutes to 16 hours. (All heating was done in a nitrogen atmosphere.) To minimize any composition changes, the toroids were annealed inside hollowed-out blocks of similar material, and completely surrounded by powder of the same composition.

C. MAGNETIC MEASUREMENTS

Measurement of magnetic properties such as saturation magnetization, remanence, initial permeability, and coercivity were made in the ways described in an earlier report[1].

III. RESULTS

A. SATURATION MAGNETIZATION

From Figure 1 it may be noted that the recovery behavior of ferrite toroids is markedly affected by sample thickness.

The thickest toroids, of 0.018 inch thickness, show very little change in properties on annealing, and any change that does occur is independent of time or temperature within the ranges shown. Evidently these toroids have such a small proportion of damaged material that any systematic time or temperature effects are lost in the experimental scatter.

The thinnest samples of 0.004 inch thickness, by contrast, exhibit very significant property changes on annealing, though the level of recovery seems to depend only on temperature, time being quite unimportant in the range considered. This statement seems true, even allowing for considerable experimental scatter.

Intermediate thickness (0.008 inch) samples do seem to show a slight sensitivity to time of anneal, but again recovery is basically temperature controlled.

B. COERCIVITY

Coercivity measurements are plotted in Figure 2, from which it can be seen that recovery effects are the exact inverse of those observed for saturation magnetization, in the sense that H_C decreases when B_S increases.

C. REMANENCE

Remanence measurements plotted on Figure 3 are in one respect different from the results of saturation magnetization plots. Even in the thickest samples a marked recovery of B_r is evident, again largely independent of annealing time.

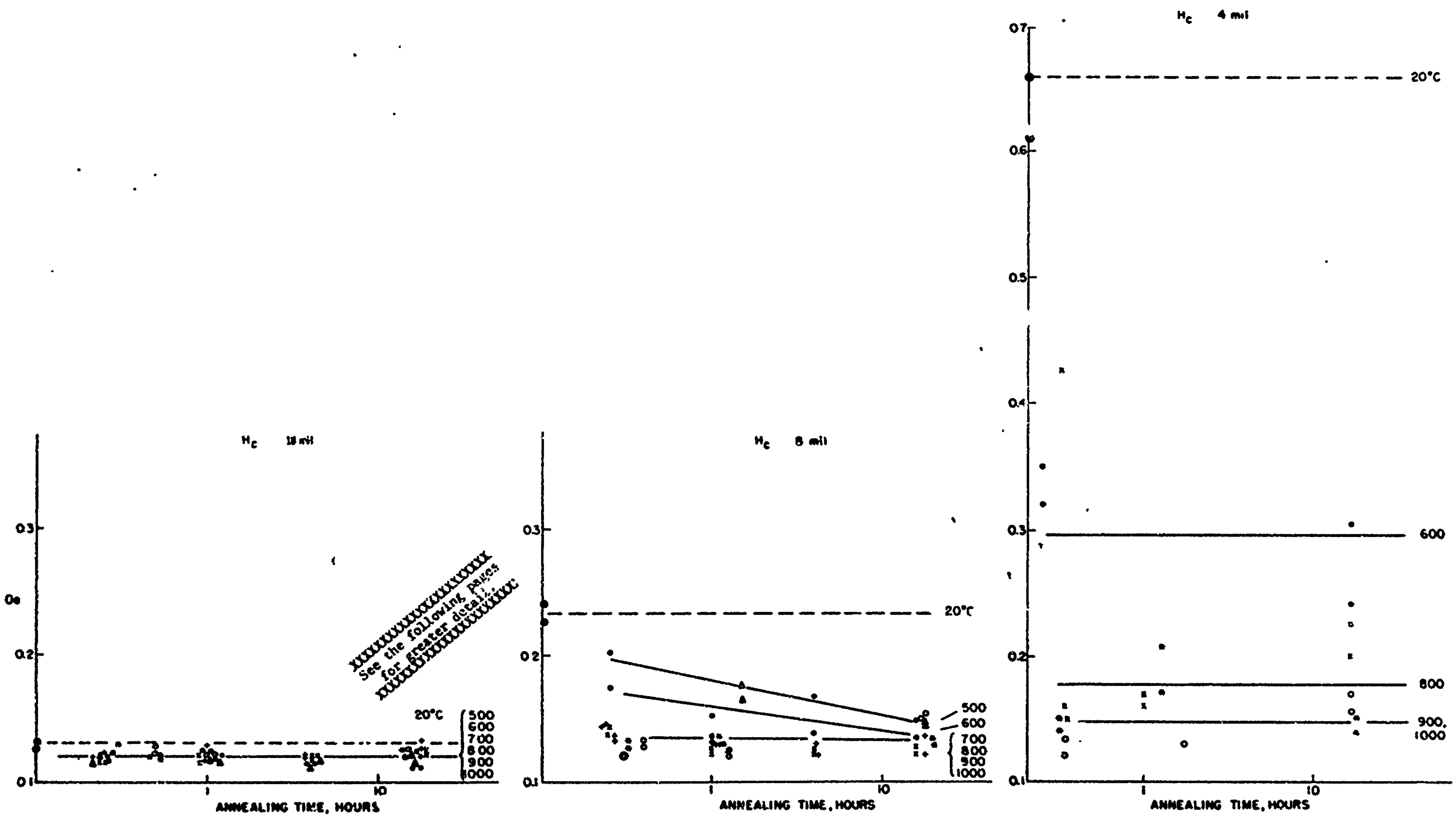

Figure 1. Effects of Annealing upon the Coercive Force of Ferrite Toroids

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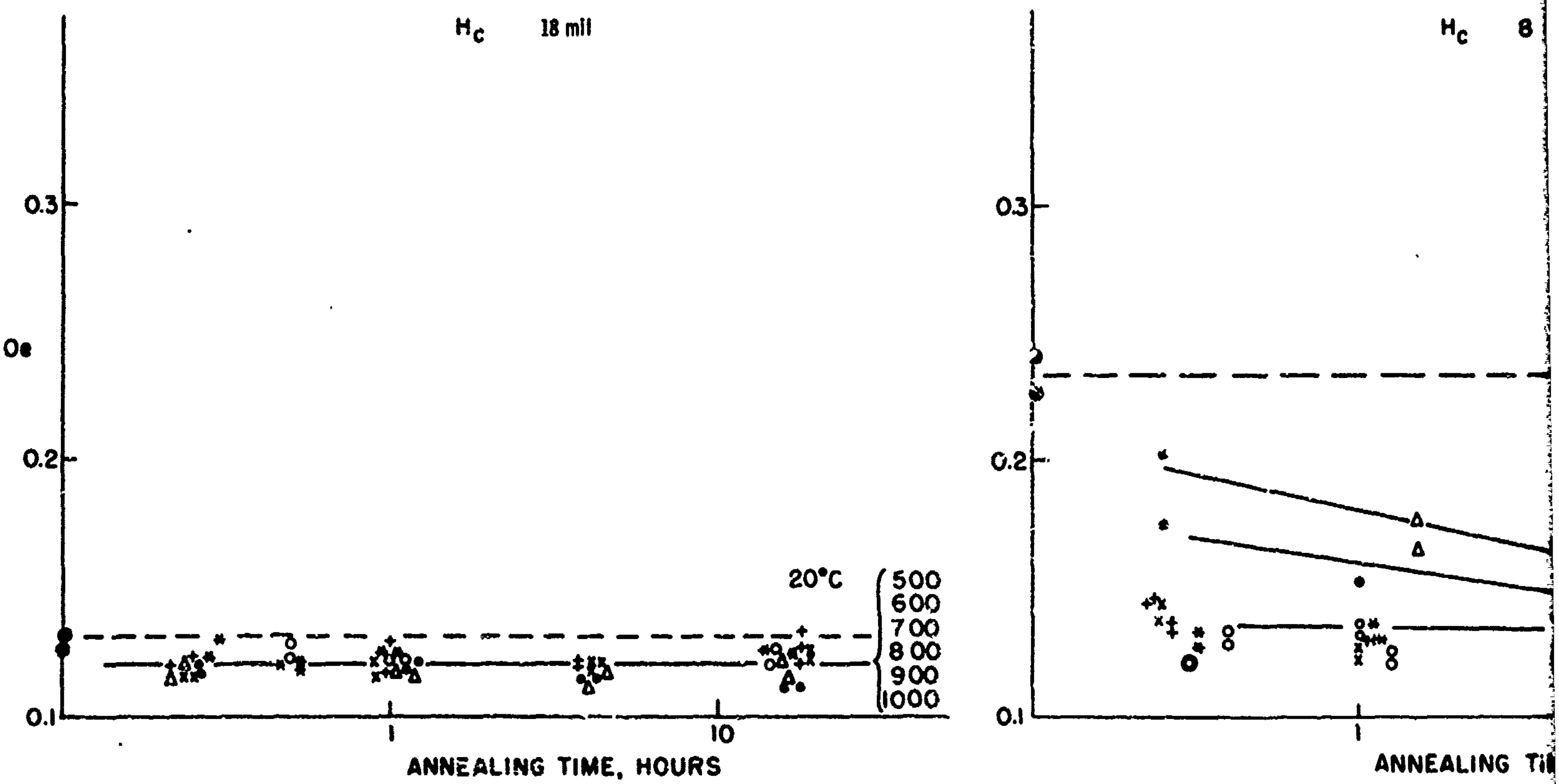

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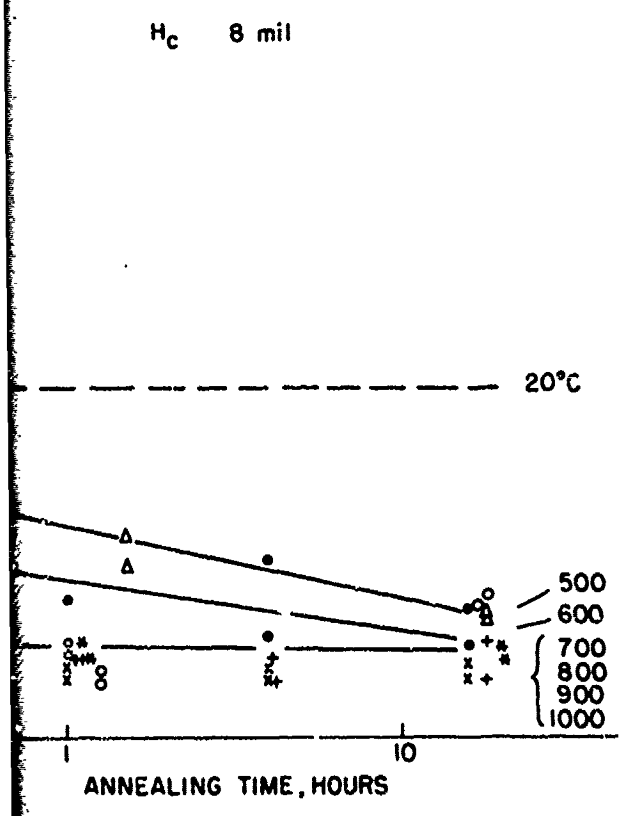

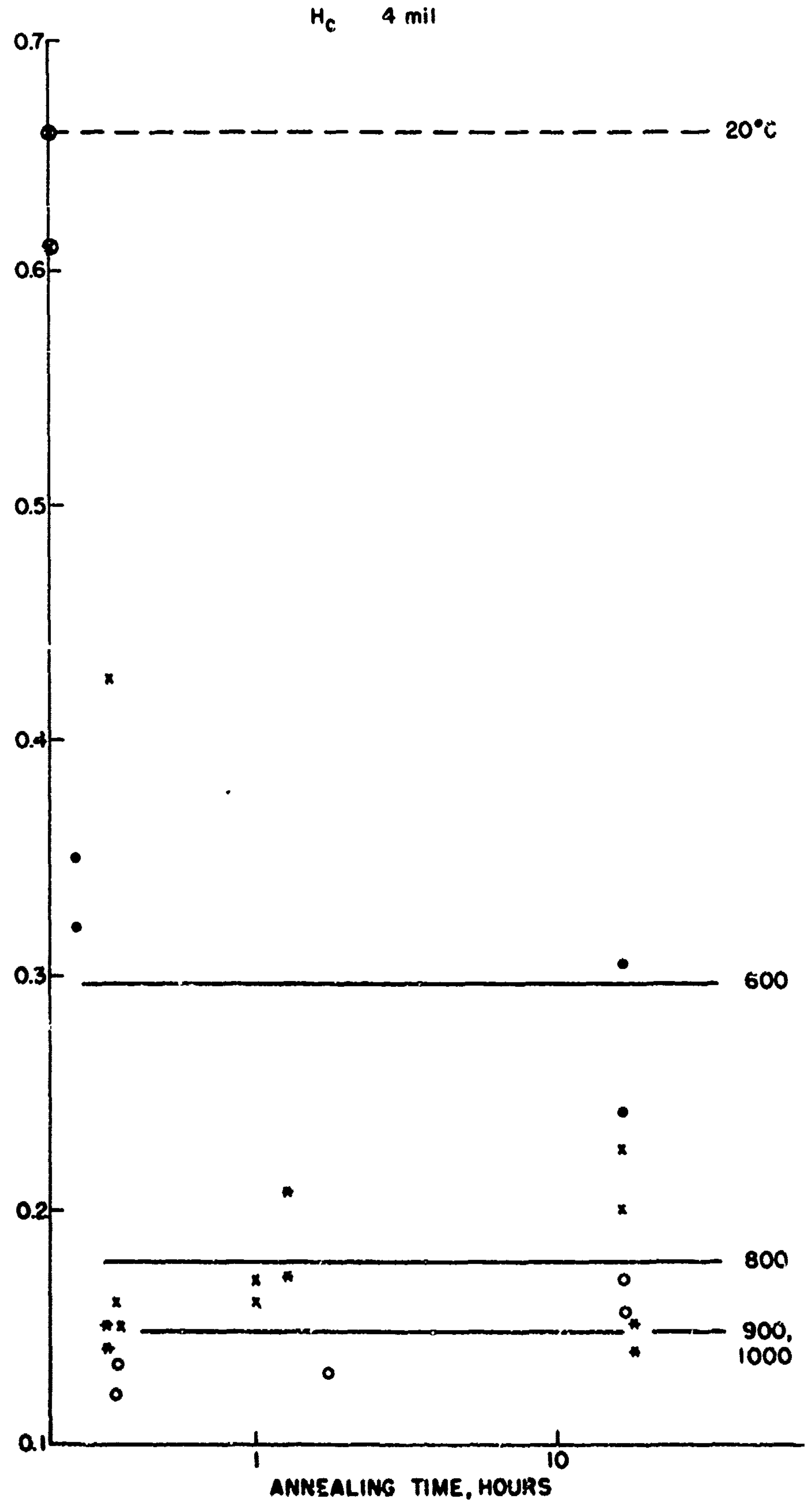

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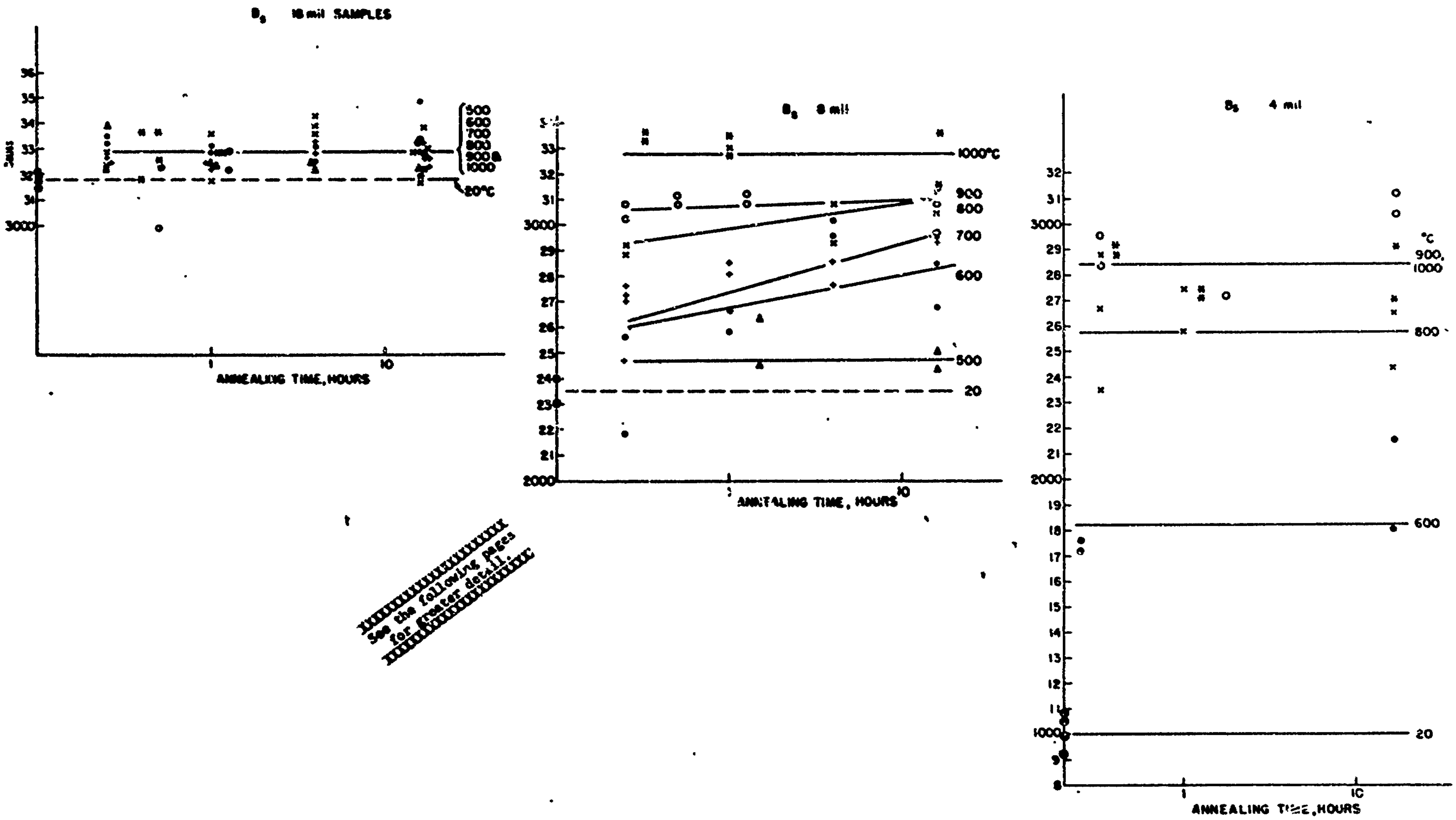

See the following pages for greater detail.

Figure 2. Effects of Annealing upon the Saturation Magnetization of Ferrite Toroids

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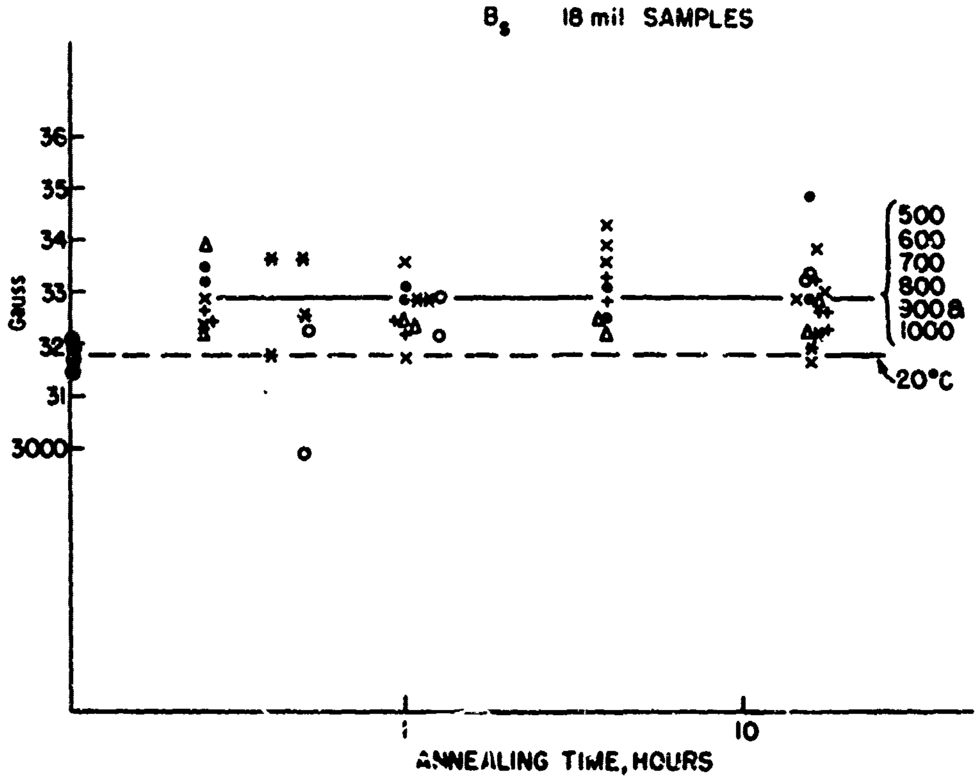

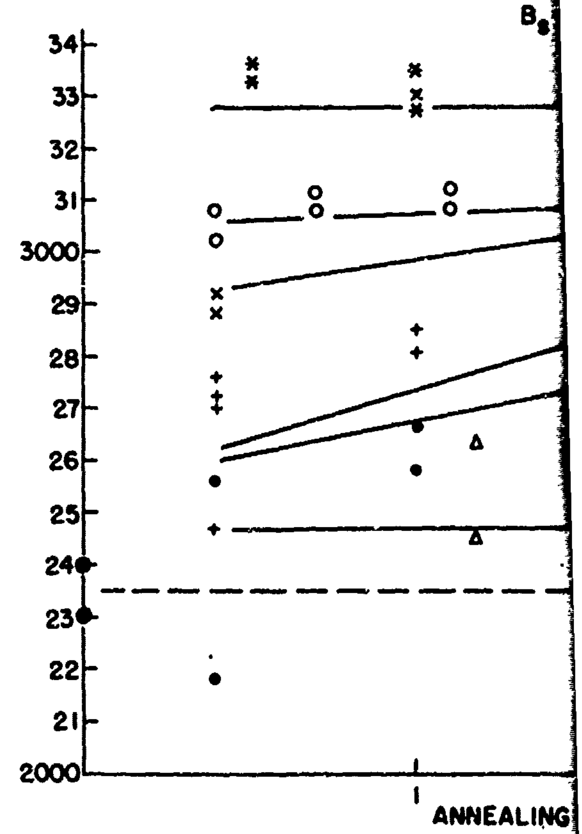

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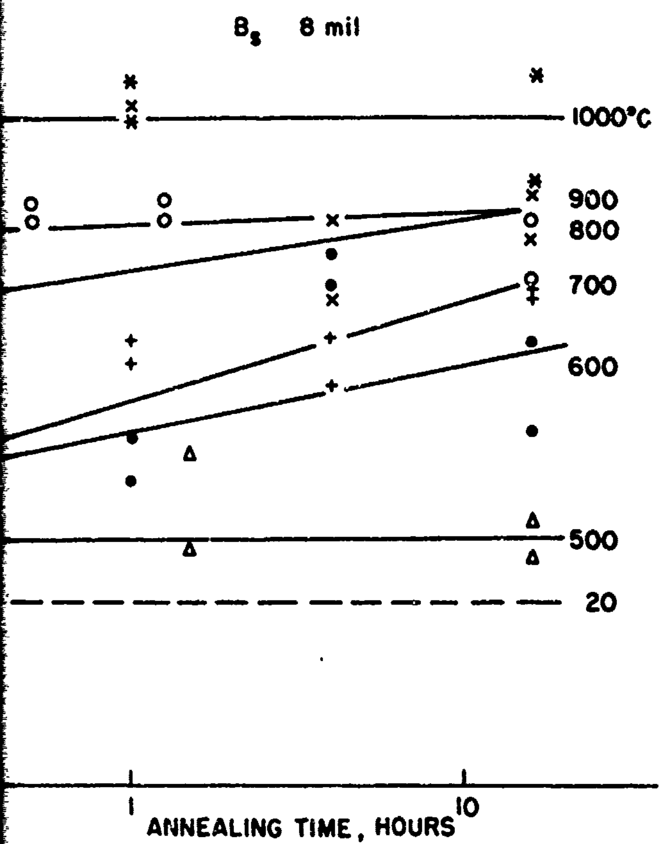

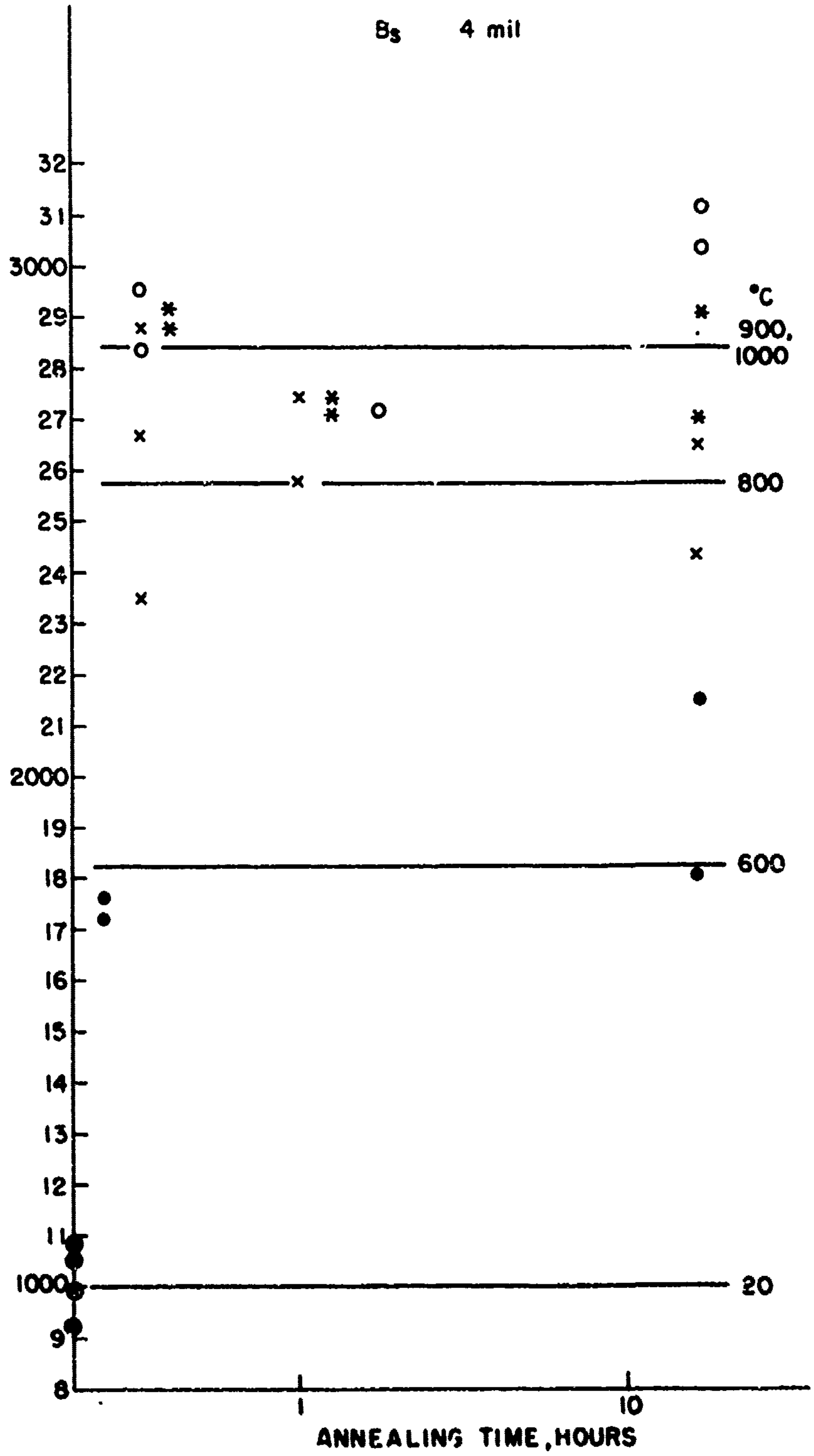

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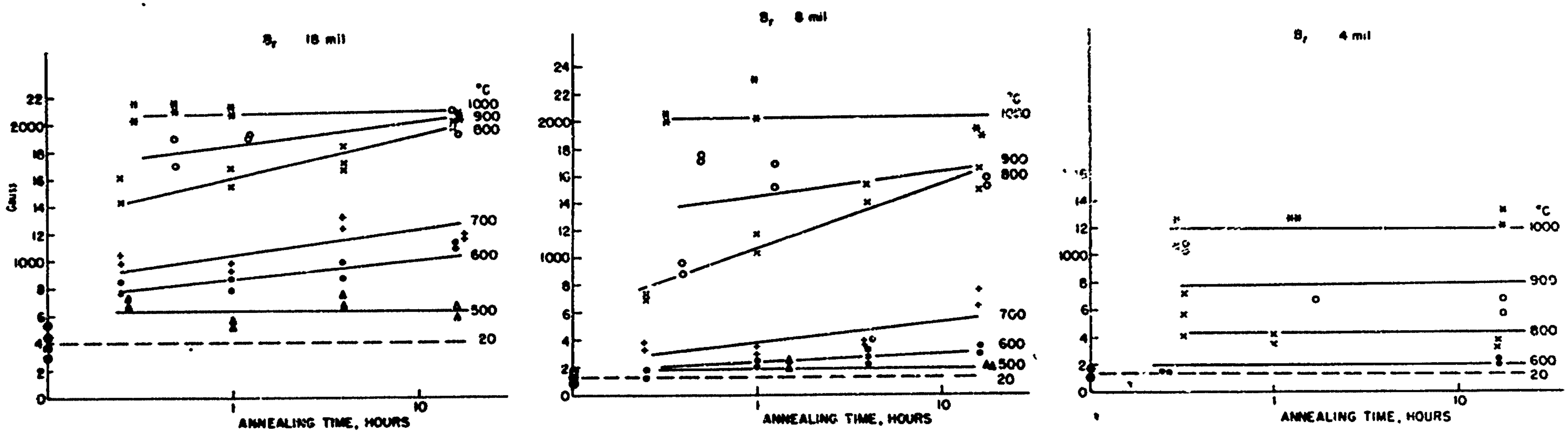

Figure 3. Effects of Annealing upon the Remanence of Ferrite Toroids

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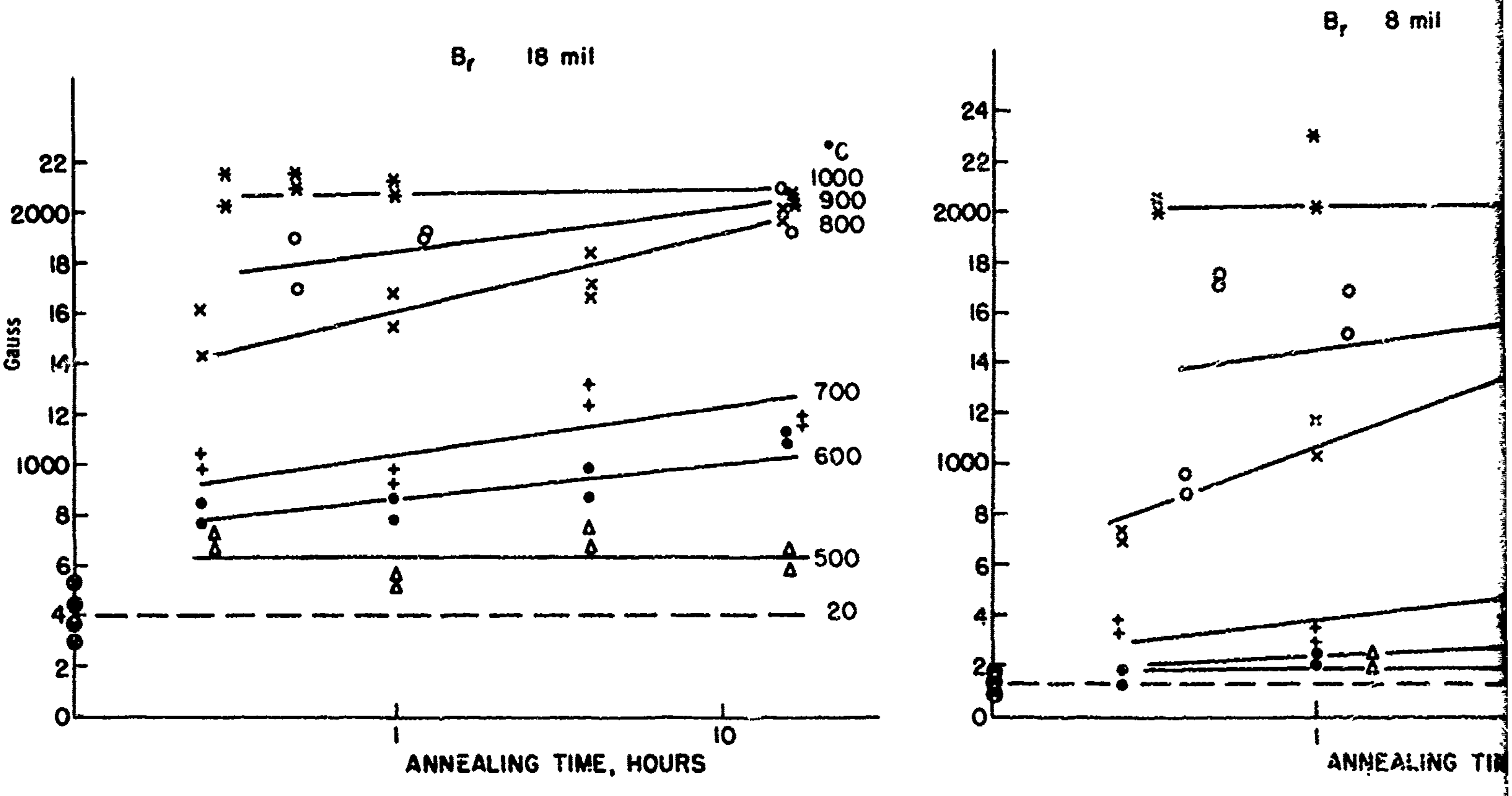

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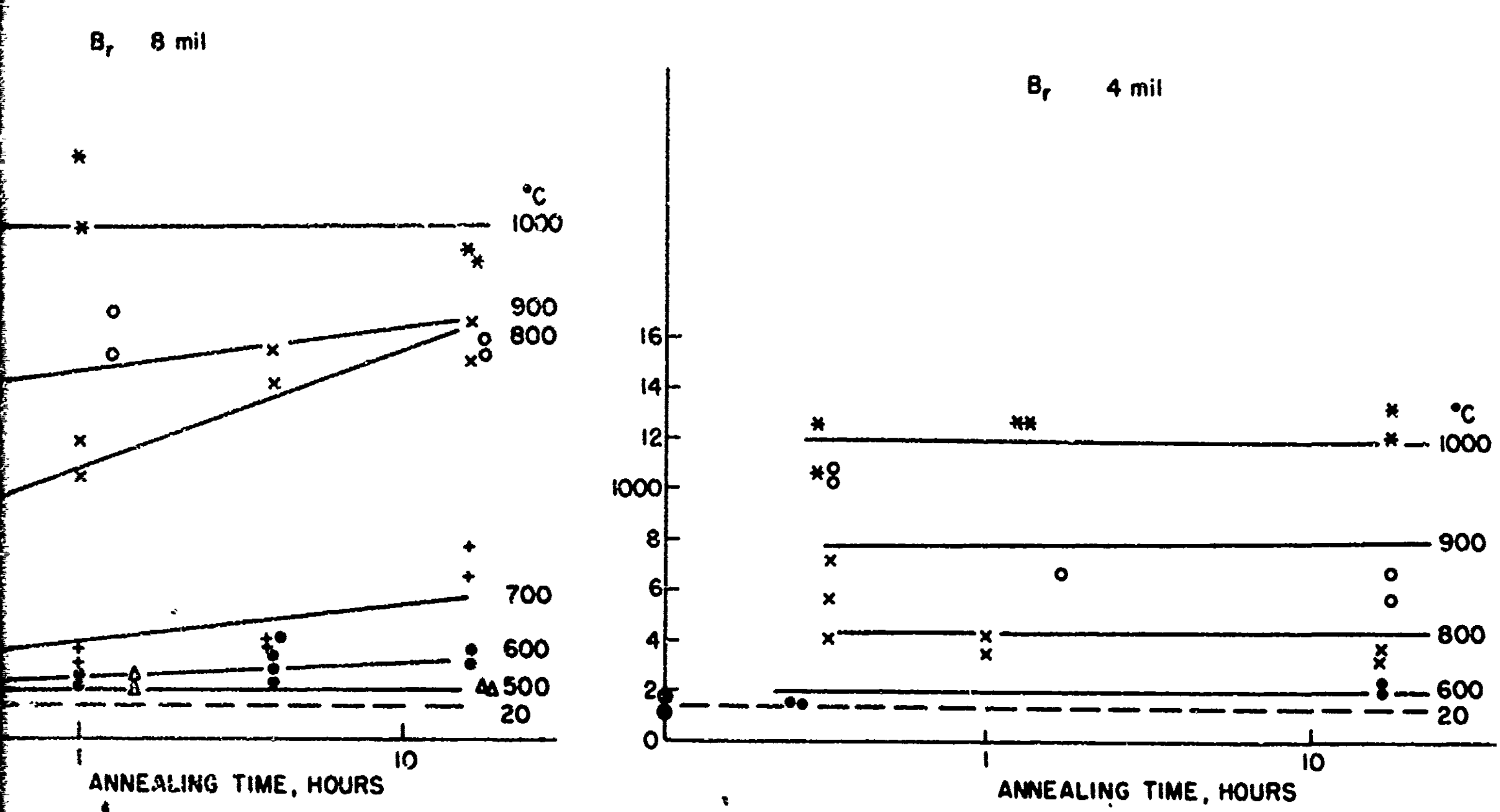

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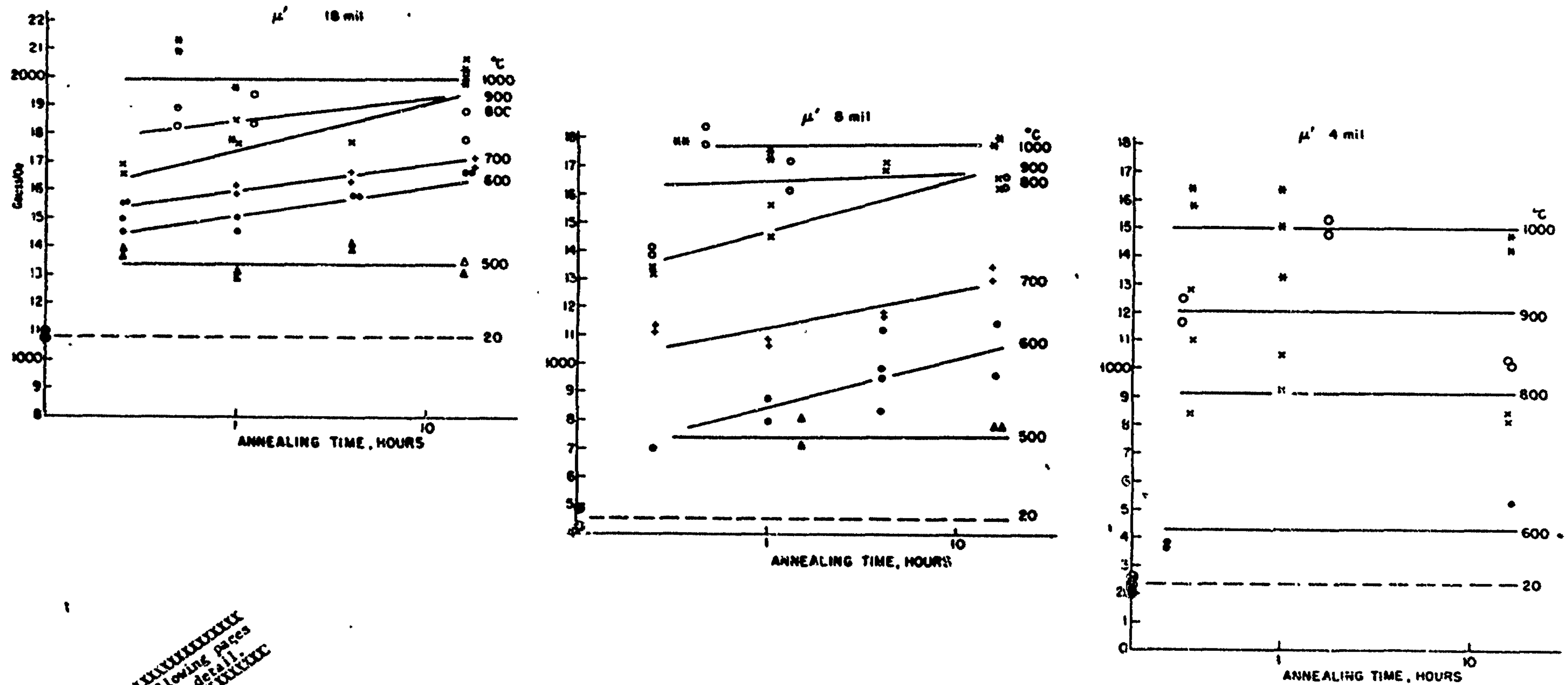
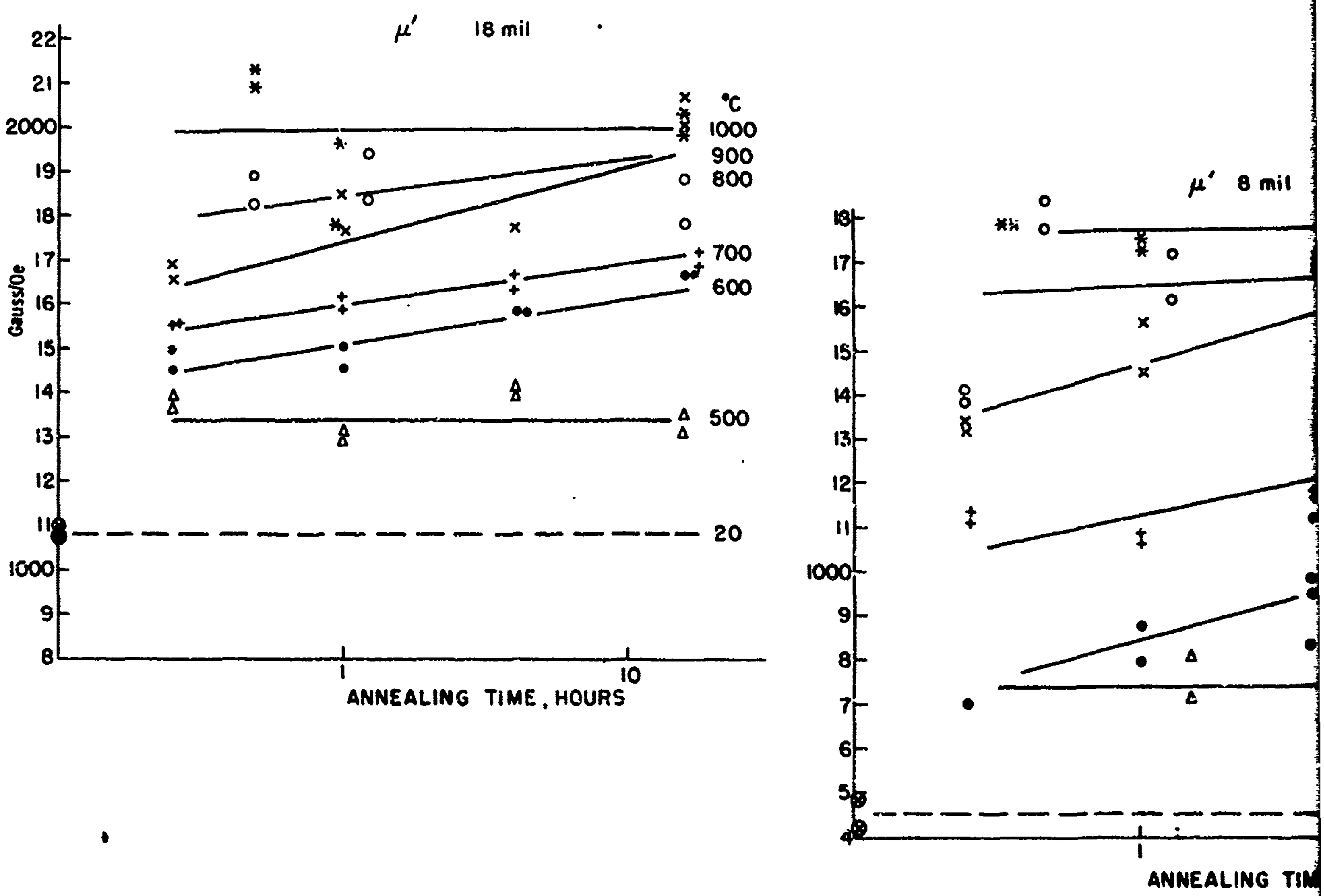


Figure 4. Effects of Annealing upon the 200 KHz Permeability of Ferrite Toroids

See the following pages for greater detail.

8.1

8.2



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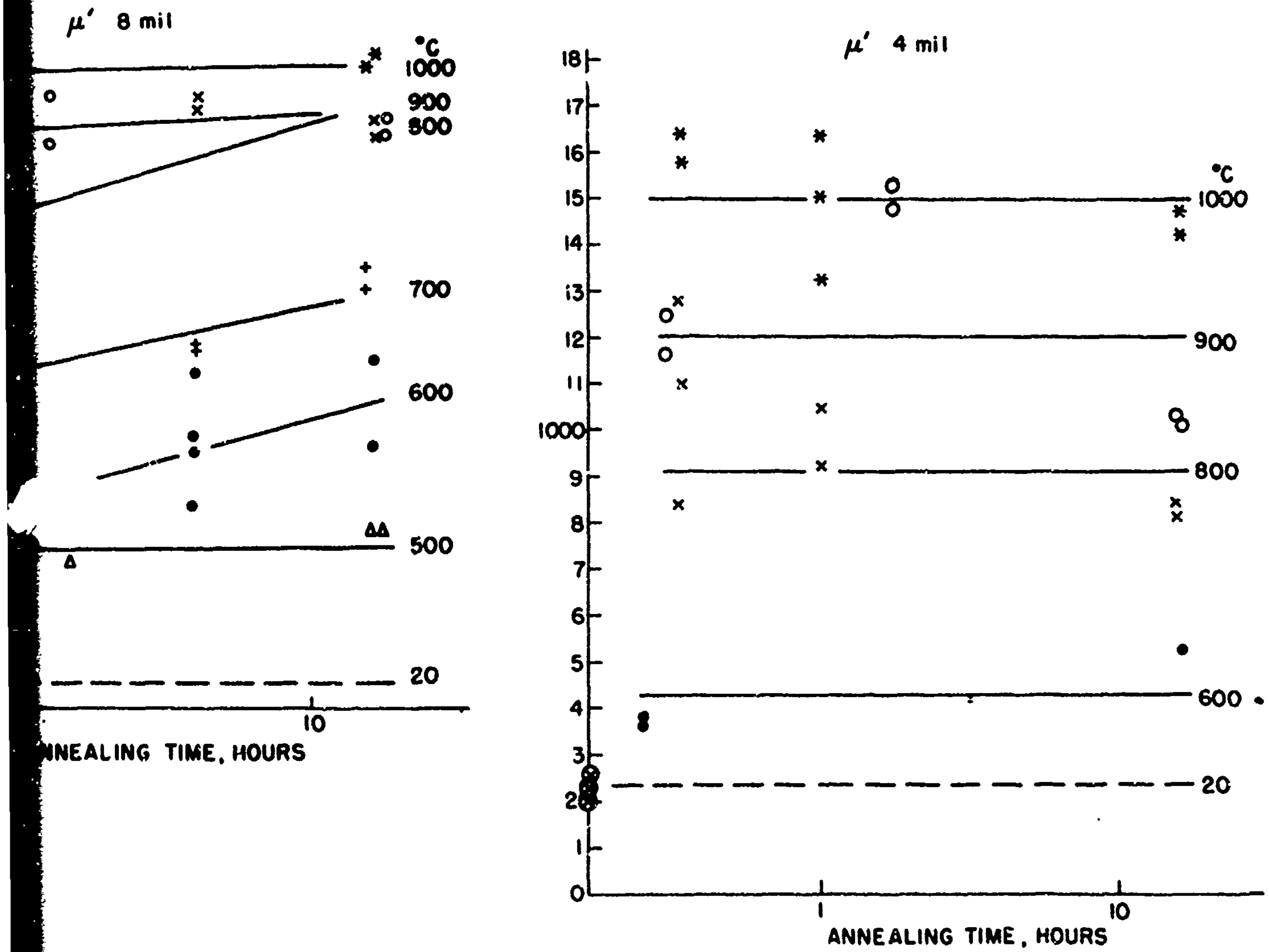

00 KHz Permeability of Ferrite Toroids

8.2

D. INITIAL PERMEABILITY AT 200 KHz

Permeability results shown in Figure 4 follow closely the pattern set by remanence measurements. Except for the thickest samples, recovery effects appear to be similar to those encountered in the saturation magnetization tests.

IV. DISCUSSION OF RESULTS

In considering the recovery of magnetic properties by annealing, it may be worthwhile to recall the way in which those properties were initially degraded by polishing. Briefly, to recapitulate a previous report [1], it was found that polishing effects could be rationalized in terms of a stress-induced phase change in surface material together with residual elastic stresses in undamaged interior material. A surface layer of about 0.002 inch thickness was postulated to contain a large concentration of stacking faults, the rest of the toroid containing residual stresses.

It was further noted that twc classes of properties existed; extensive properties, such as B_S and H_C, sensitive only to phase change, and intensive properties like μ' and B_r which are stress-sensitive.

The present annealing results seem in general to be compatible with earlier suggestions, since recovery effects clearly distinguish two classes of properties. B_S and H_C do not show any marked changes during annealing of thick samples (i.e., basically undamaged material containing only residual stresses), whereas B_r and μ' do show such changes. It may therefore, be inferred that B_r and μ' are stress-sensitive properties, while B_S and H_C are not.

We now face a seeming paradox. Granted that the results on thick samples seem to confirm an earlier hypothesis that stacking faults and residual stresses are created by polishing, why is it that results from thin samples show no distinction between stress-sensitive and phase-sensitive properties? In thin samples recovery is always athermal, or time independent, a circumstance which is most unusual in stress relaxation. Assuming that stress relaxation can occur by vacancy diffusion leading to dislocation climb, one would expect the process to be quite rapid at a temperature around $T_m/2$ where T_m is the absolute melting point of the material. In the case of ferrite, a melting point around 1600°C seems reasonable, leading to the conclusion that thermally-activated processes should predominate during anneals carried out abo\ -700°C.

The fact that such thermally-activated stress relaxation processes do not seem to be important in thin samples, even for stress-sensitive properties like μ' and B_r, leads one to suppose that any stresses present are not free to relax in the way that random dislocation tangles due to cold-work are.

It might be, for example, that coherence strains due to stacking faults are locked in until the stacking faults are themselves dissolved. Such an explaration might neatly resolve our paradox, since strains (and hence stresses) would not be independent of phase changes. One could then argue that stacking faults are metastable, and that their metastable equilibrium proportion is temperature dependent in the same way as stable precipitates in systems exhibiting marked solubility variations with temperature -- like many precipitation-hardening alloys.

On this basis, annealing effects might be primarily due to the resorption of stacking faults with a concurrent relaxation of coherency strains.

Clearly then, the process could be virtually athermal, and in samples containing large amounts of second phase both classes of property, the phase-sensitive and the stress-sensitive, would behave alike.

In conclusion, one further aspect of this work may be discussed, whether or not the present results could be of practical usefulness in a sensor.

It seems clear, for example, that 0.004 inch thick polished toroids could be used to indicate the maximum temperature to which they might be exposed since by monitoring the initial permeability, say, it should be possible to differentiate a 700°C excursion from a 600°C one. Furthermore, the useful range could extend to above 1000°C. It has to be emphasized, however, that the present results are of limited interest until a quantitative model of the damage and recovery effects can be developed. Such an understanding does not exist at the moment, and will require considerable effort to develop.

It will need extensive transmission electron microscopy with high temperature diffraction capability, extensive magnetostriction measurements, and much mathematical modeling to achieve worthwhile results. At this moment, the topic can only be regarded as a mildly interesting curiosity.

V. CONCLUSIONS

Magnetic property changes in nickel-zinc ferrite toroids caused by polishing can readily be recovered by annealing at temperatures up to at least 1000°C.

Recovery appears to be an athermal process, leading to speculation that internal strains (and hence stresses) may be due to coherency effects between stacking faults and the parent ferrite lattice; thus, stress-relief is not dependent of the presence of the postulated second phase.

The possibility of using recovery effects to indicate maximum temperature exposure in some type of sensor is considered.

REFERENCE

1. "Effects of Cold-Work On The Magnetic Properties of Nickel-Zinc Ferrite", J. R. Kench, 5th Technical Report, Office of Naval Research Contract No. N00014-69-C-0123, June 1972.